Tender Energy X-ray Spectroscopy (TES):
A beamline for high performance spatially resolved X-ray absorption spectroscopy and imaging at mm to micron scales, optimized for the "tender" energy range from 1 up to 8 keV

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Overview of TES capabilities:

- Energy range 1-8 keV
  - Optimized for *Tender* 1-5 keV range
- Spot size user-tunable from 1 mm to 1 μm
- Microbeam flux up to $2x10^{12}$ ph/sec at sample
- Microprobe: extend XRF imaging and XAS capabilities to lighter elements
- Optimized for high-performance XAFS at all available spot sizes
- Helium glove-box sample environment
- On-the-fly scanning of either stage or monochromator

Tender energy range:

- Access K edges of lighter elements…
  - Mg, Al, Si, P, S, Cl, Ca, Ti
- …and uniquely advantageous L and M edges
  - Cd, Sb, Zr, W, Mo, Th, U, Pu
  - Sensitivity to oxidation state and coordination
- Requires specialized design of entire beamline
  - Optics, monochromator crystals, windows
- Helium-atmosphere sample environment
  - Wet or vacuum-sensitive samples *in their native states*
- Specialized fluorescence detectors
  - Low-energy sensitivity, compatibility with He atmosphere

XRF imaging

Why you need a low-energy incident beam even for XRF:

- Light elements have low cross-section at high energy

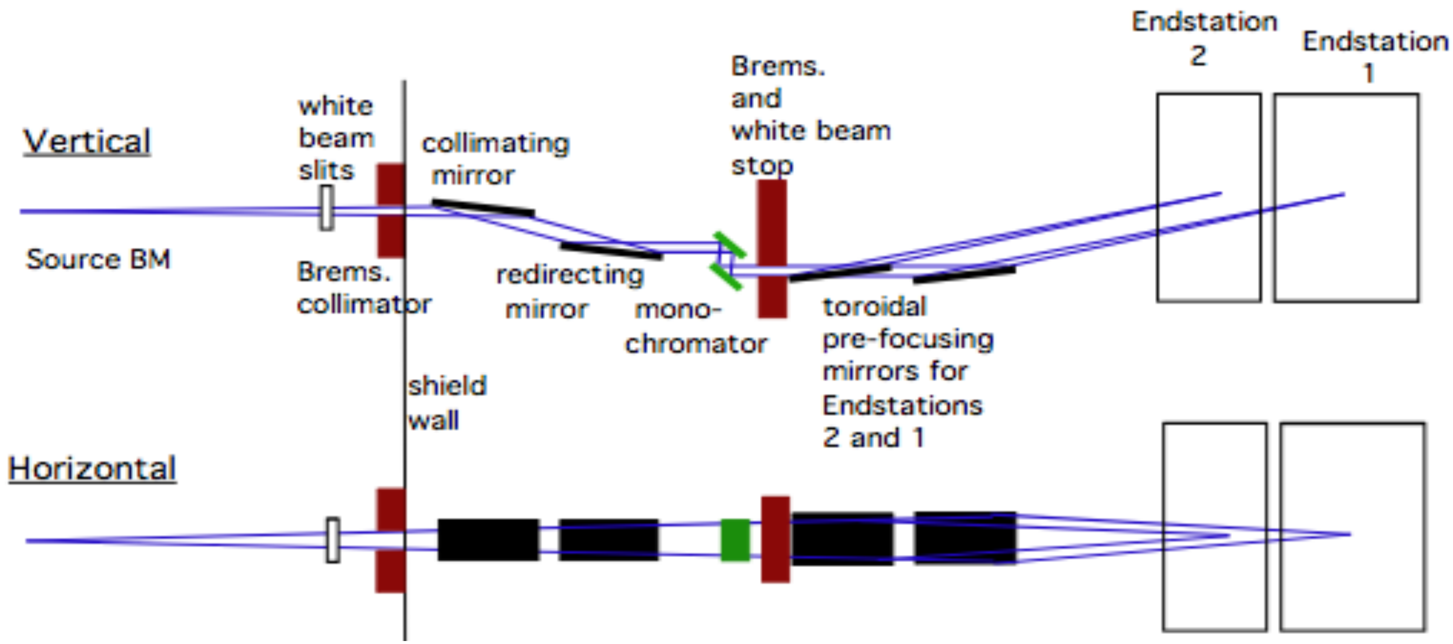
Measurable fluorescence counts produced by a hypothetical sample as a function of incident beam energy.	Incident energy	Na	Mg	Al	Si	P	Fe
	at abs. edge	1000	1000	1000	1000	1000	1000
	at 2 keV	214	378	575	822	--	--
	at 3 keV	71	124	200	308	455	--
	at 5 keV	16	30	50	76	159	--
	at 10 keV	2	4	7	11	15	427

- To see low-concentration light elements that would be masked by fluorescence from slightly heavier elements at high concentration

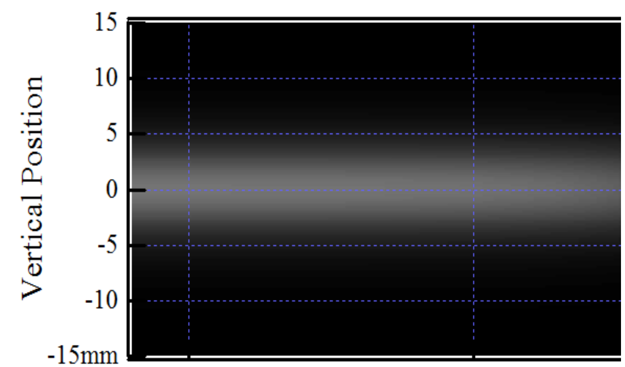
Spatially-resolved XAFS:

- XANES to full-length EXAFS scans at microbeam resolution
  - Heterogeneous or structured materials
  - Tune beam size to experimental needs
  - Measurements of single particles
- Requires optimization of beamline design for XAS
  - Stable energy scanning and repeatability (within 0.1eV)
  - Stable beam position over 1000eV span (35° of mono)
  - High flux and detector sensitivity for low concentrations
- XAS imaging in two modes:
  - Image stack at increments of energy
  - Energy scan at each pixel (fast scanning of mono)

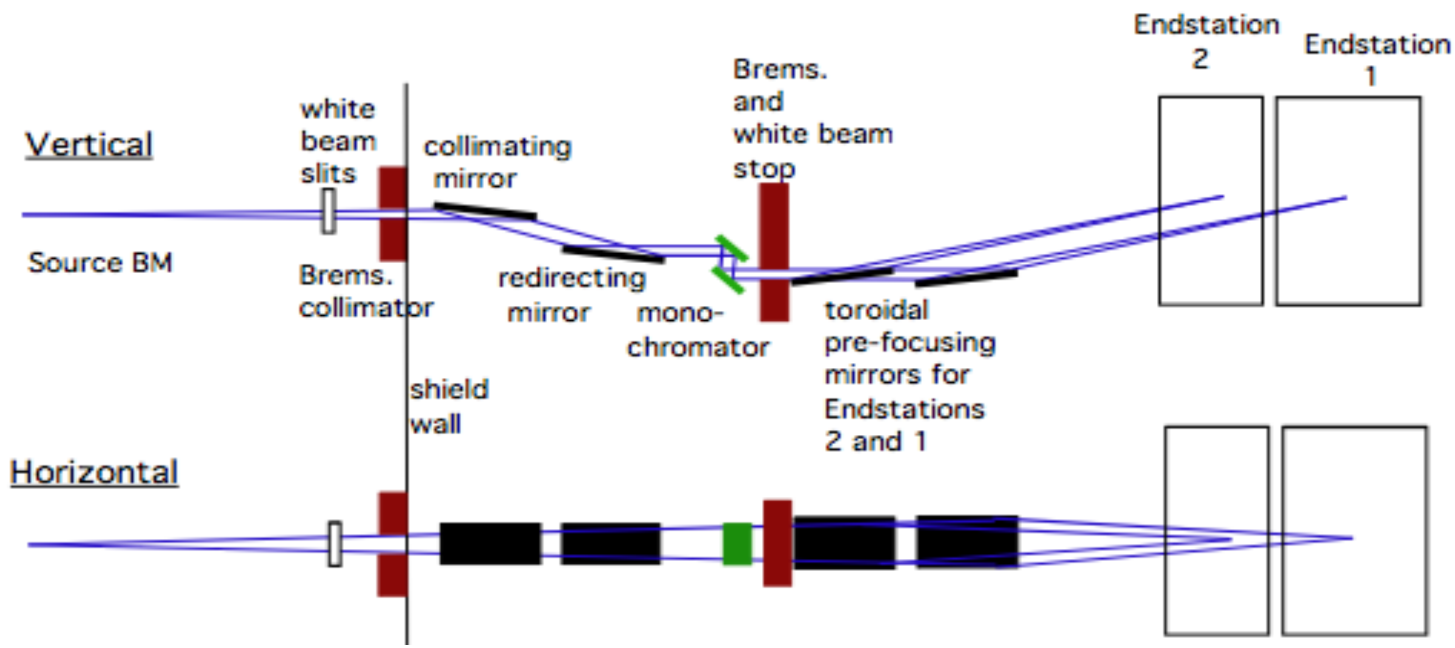
TES Beamline layout:



- Source: NSLS-II dipole bend magnet is ideal for TES
  - Ec=2.39 keV, smooth broadband spectrum over energy range, small source size for focusing, no spatial or energy-dependent structure, high brightness.

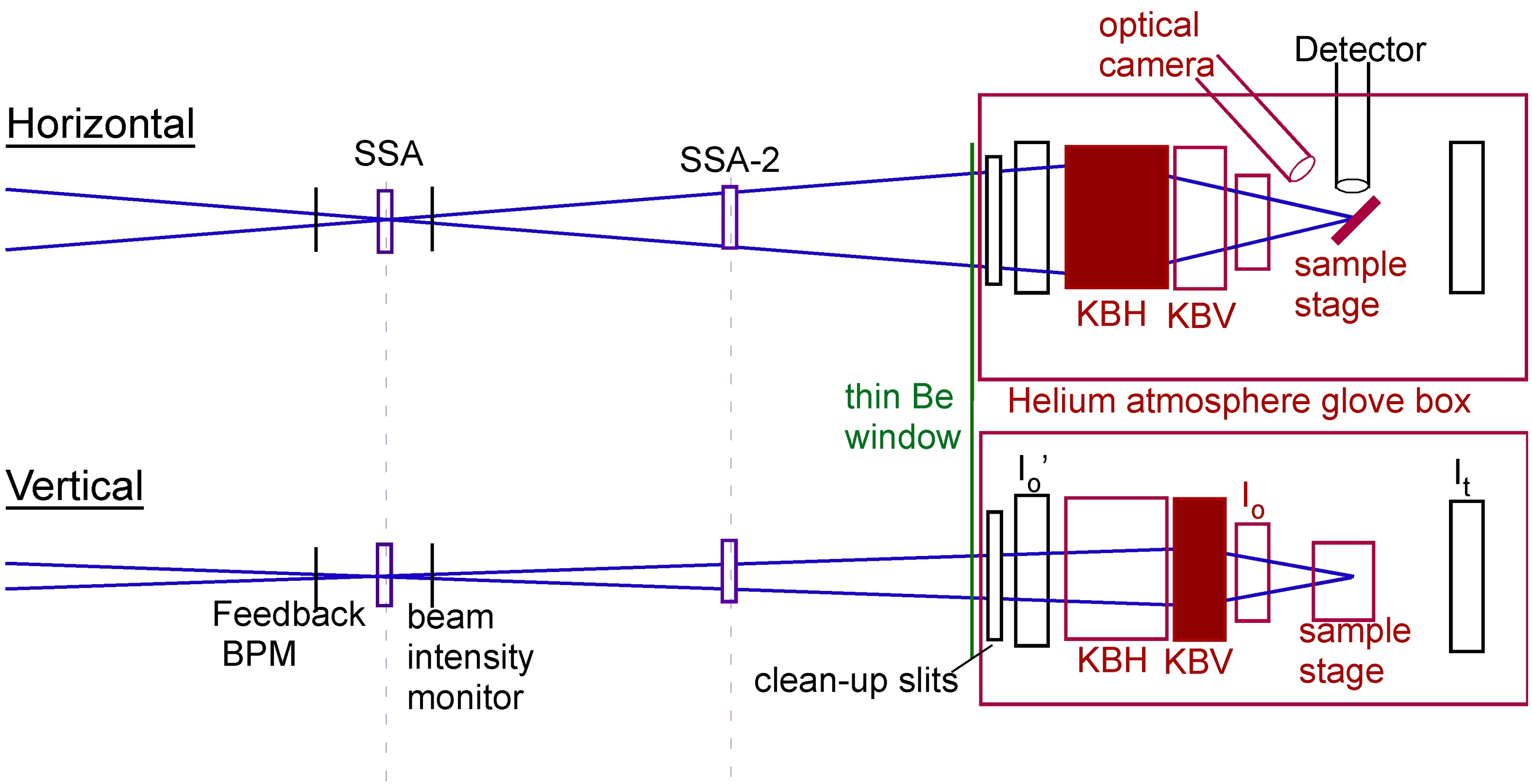


TES Beamline layout:



- Collimating/harmonic-rejection mirror, paired with redirecting mirror, adjustable pitch 6-26 mrad
- Fixed-exit monochromator, Si(111), InSb, Beryl…
- Macro-focusing toroidal mirrors
- Two endstations: microprobe and bulk/*in-situ*

Microprobe endstation:



- Secondary source aperture at macro focus
- KB mirrors in sample chamber (He glove box)
- Spot size tunable by adjusting SSA (H and V independent)

Microprobe endstation:

- Operation as a He-atmosphere glove box
- Sample stage similar to hard X-ray microprobes
  - Standard 45° geometry
  - Accommodate large samples or *in-situ* cells
- Detectors for low and high count rates
  - Ultra-low-energy Ge detector, Vortex Si detector
  - Either detector can extend to within a few mm of sample
- Optical camera facing sample

Performance:

- Ray tracing and flux calculations give the following estimates *delivered to sample*:

	Endstation 1	Endstation 2		
Energy (keV)	Flux, tunable from 0.2x0.2 to 1.2x0.5 mm (ph/s at sample)	Flux, microbeam 19x23 microns	Flux, microbeam 6x7 microns	Flux, microbeam 1x1 micron
1.2	1.15 $\times10^{12}$	5.4 $\times10^{11}$	2.85 $\times10^{11}$	6.8 $\times10^{9}$
2	3.5 $\times10^{12}$	2.3 $\times10^{12}$	1.0 $\times10^{12}$	2.4 $\times10^{10}$
3	2.8 $\times10^{12}$	1.9 $\times10^{12}$	7.7 $\times10^{11}$	1.8 $\times10^{10}$
4	1.9 $\times10^{12}$	1.2 $\times10^{12}$	4.5 $\times10^{11}$	1.1 $\times10^{10}$
5	1.3 $\times10^{12}$	7.2 $\times10^{11}$	2.7 $\times10^{11}$	6.4 $\times10^{9}$
7.5	5.1 $\times10^{11}$	2.15 $\times10^{11}$	8.1 $\times10^{10}$	1.9 $\times10^{9}$

- Initial performance will be reduced in order to achieve earliest availability and minimize downtime
- Beamline macro-focusing optics will be moved from NSLS X15B and subsequently upgraded to full performance

Beamline development timeline:

June/July 2010 Beamline Development Proposal (BDP) submitted, presented to Science Advisory Committee review panel

October 2010 Beamline formally approved by NSLS-II

May 2012 NxtGen Project announced, including TES

August 2012 X15B/TES microprobe endstation project funded

October 2012 NxtGen Project formal start

~mid-2014 NSLS shutdown, NSLS-II startup, commissioning of SRX

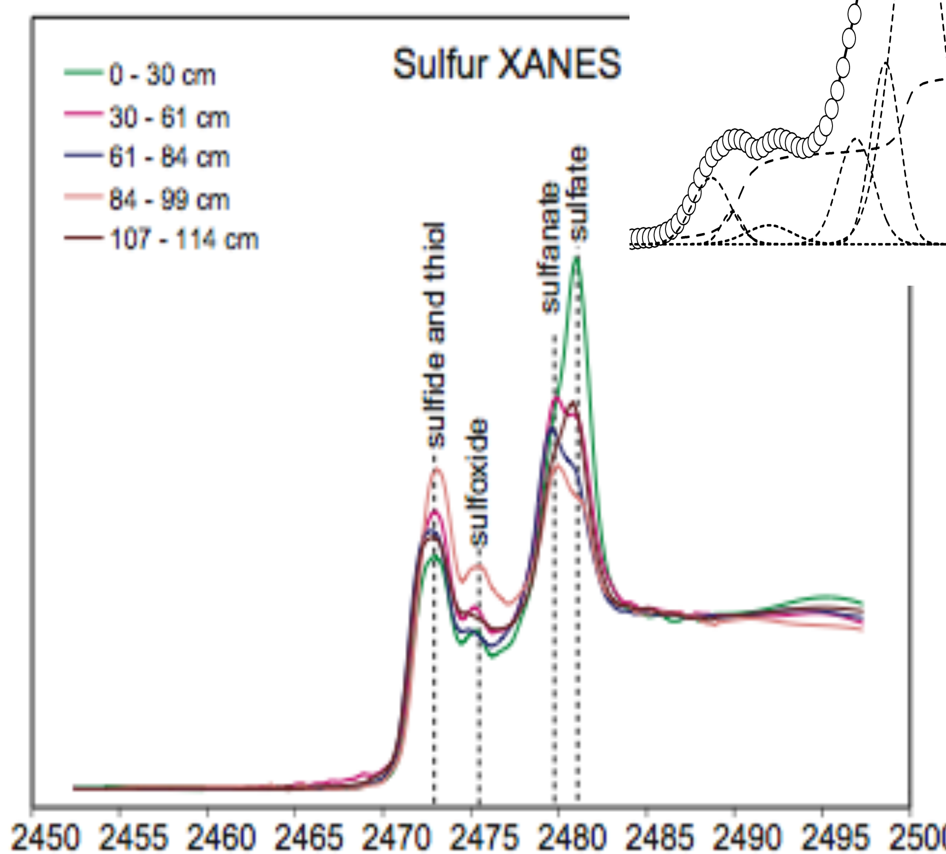
October 2014 commissioning of TES

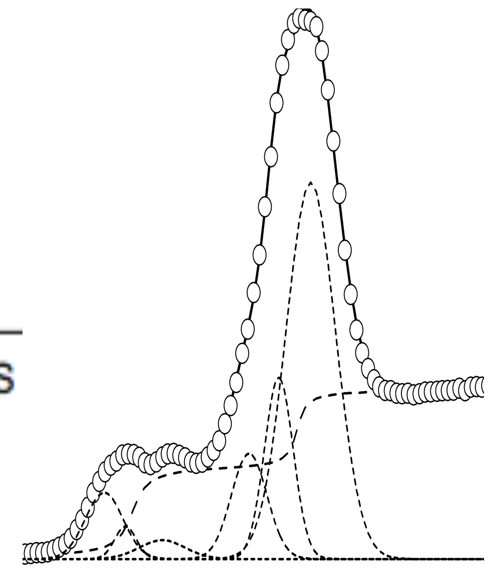
Microprobe endstation project:

- Microprobe endstation being built and commissioned at NSLS Beamline X15B now
- Jointly funded by NSF and DOE:BES through Stony Brook University
- Design mostly complete, KB mirrors and experimental chamber ordered
- Plan for commissioning February-April 2013
- **Full operation available Summer 2013**
- Move to TES mid-2014, commission October 2014

Tender XAS -- examples

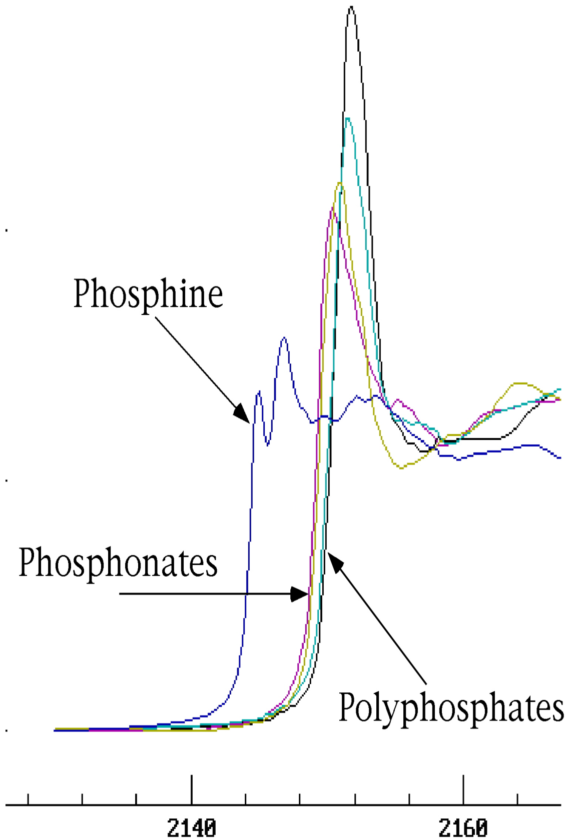
- Sulfur XANES:
  - Oxidation state, speciation
  - Sulfide, thiol, disulfide, sulfoxide, sulfonate, sulfate
  - Distinct peaks over 10 eV

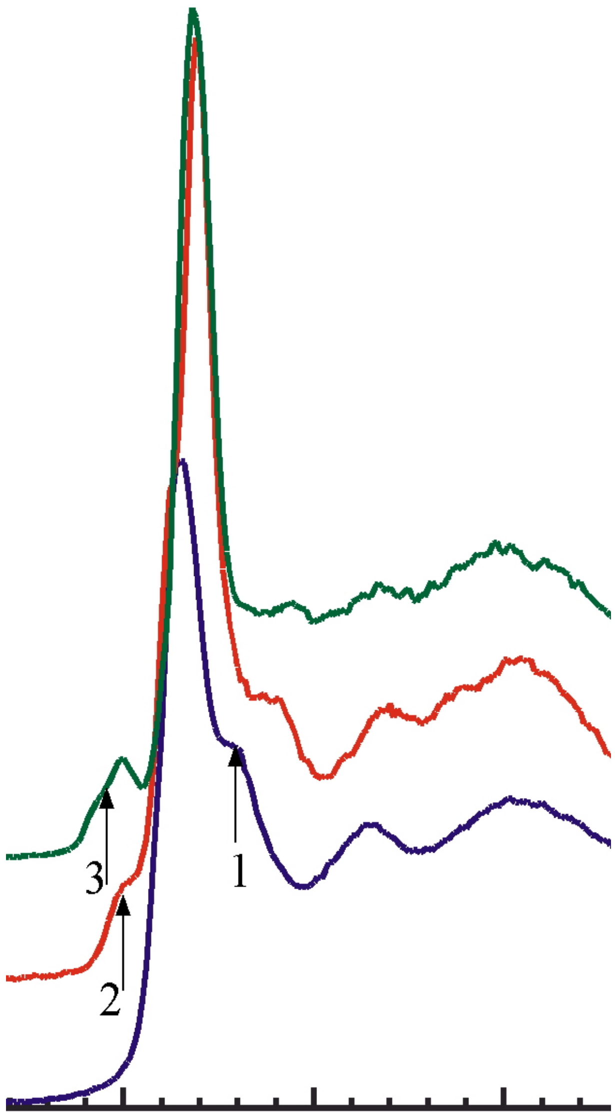




Phosphorus

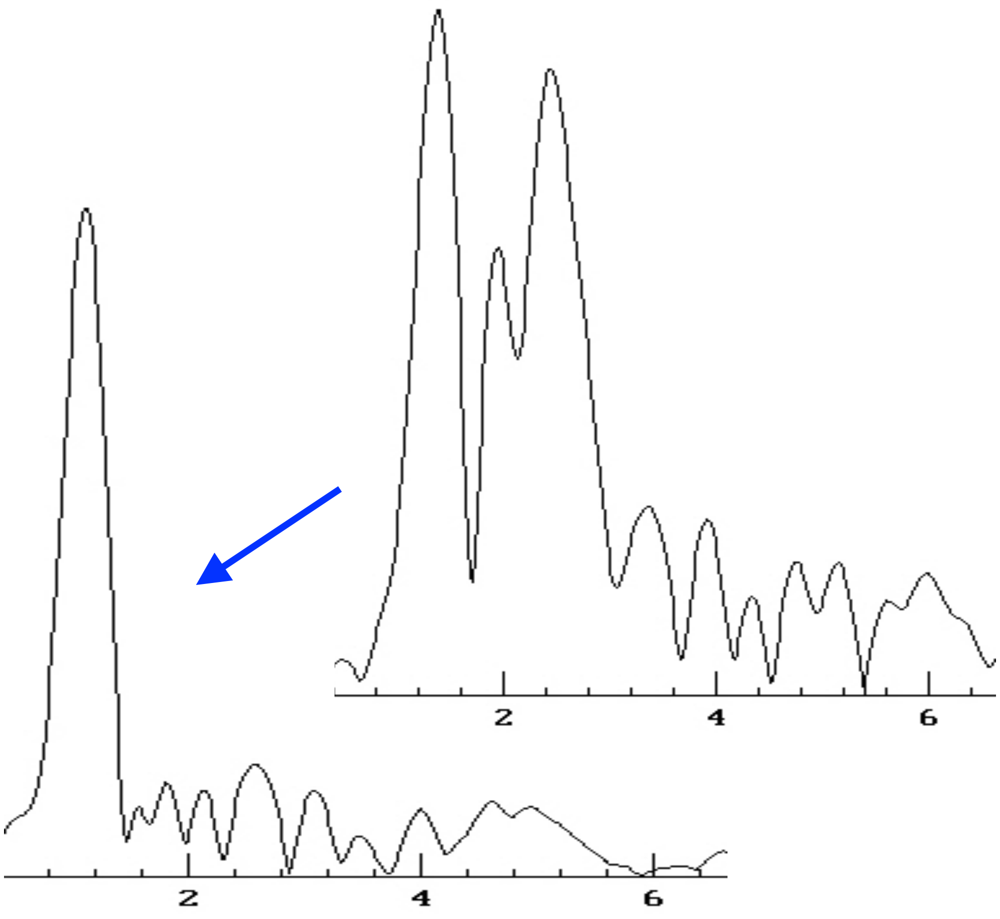
- XANES:
  - Oxidation state
  - Chemical coordination, Ca, U, Fe
  - Library of mineral standards





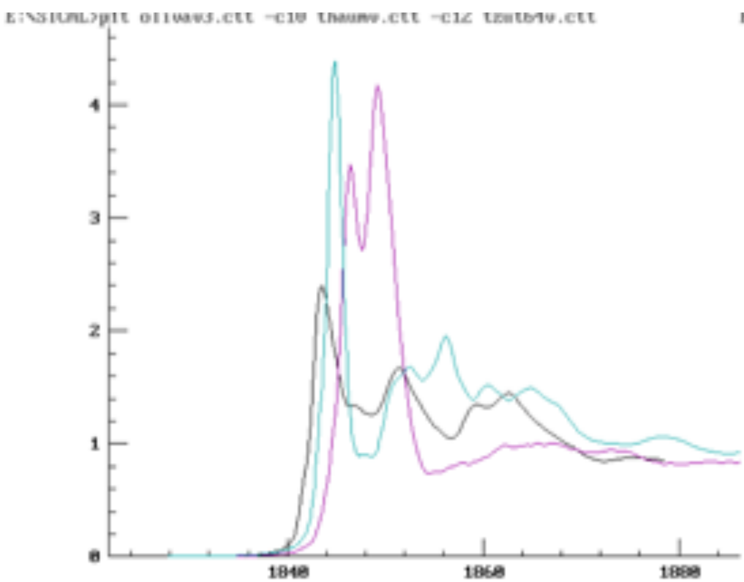
Phosphorus

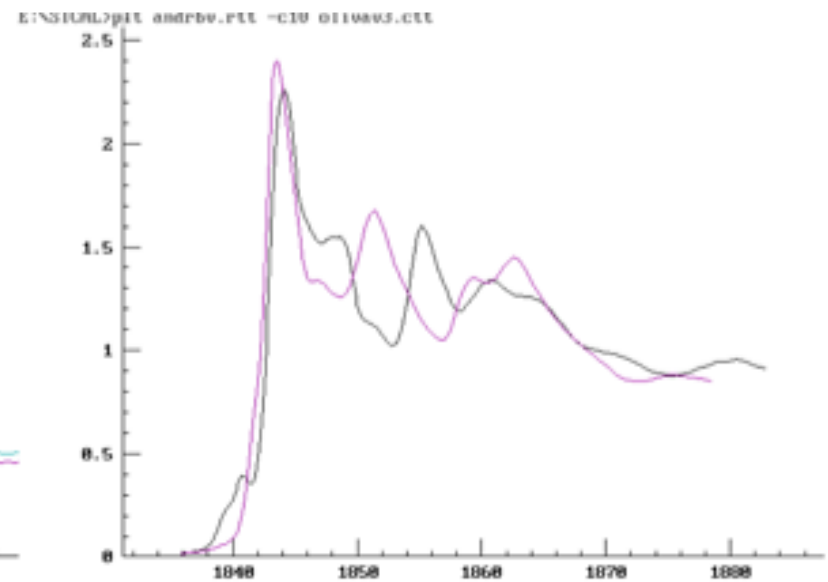
- EXAFS:
  - Local structure (out to ~8Å)
  - Example: conversion of organic phosphate ester to free phosphate by microbial phosphatase

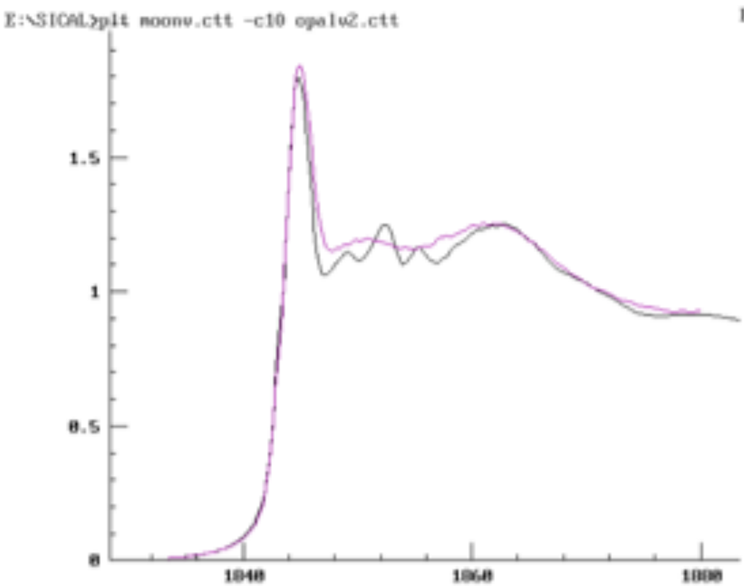


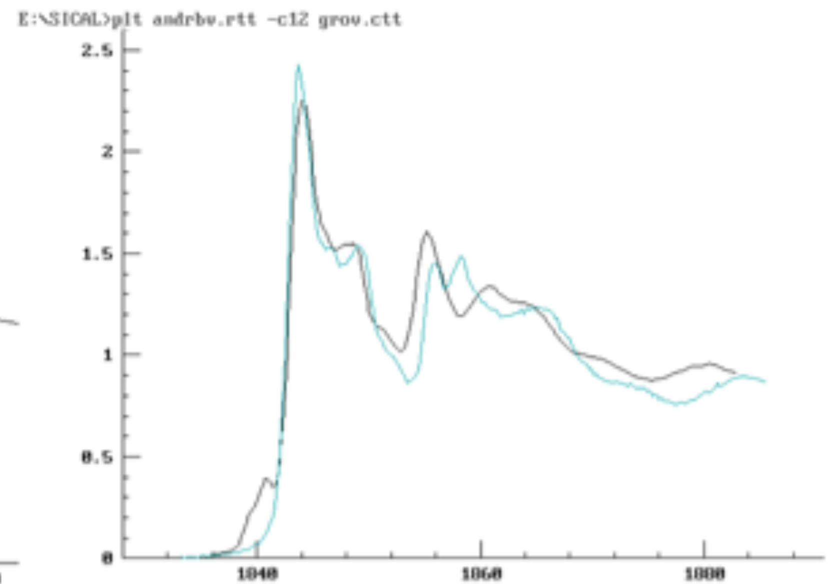
Silicon

XANES



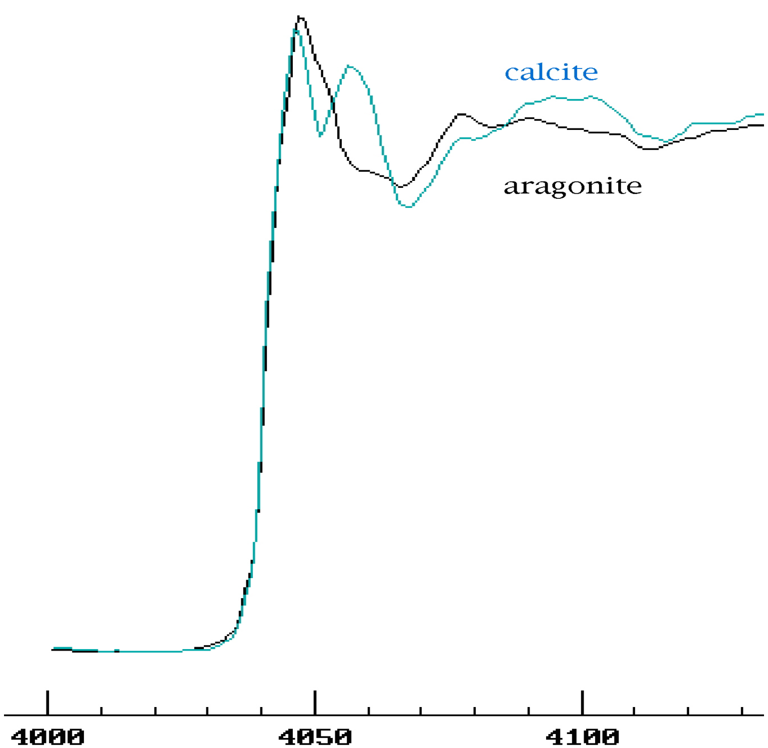


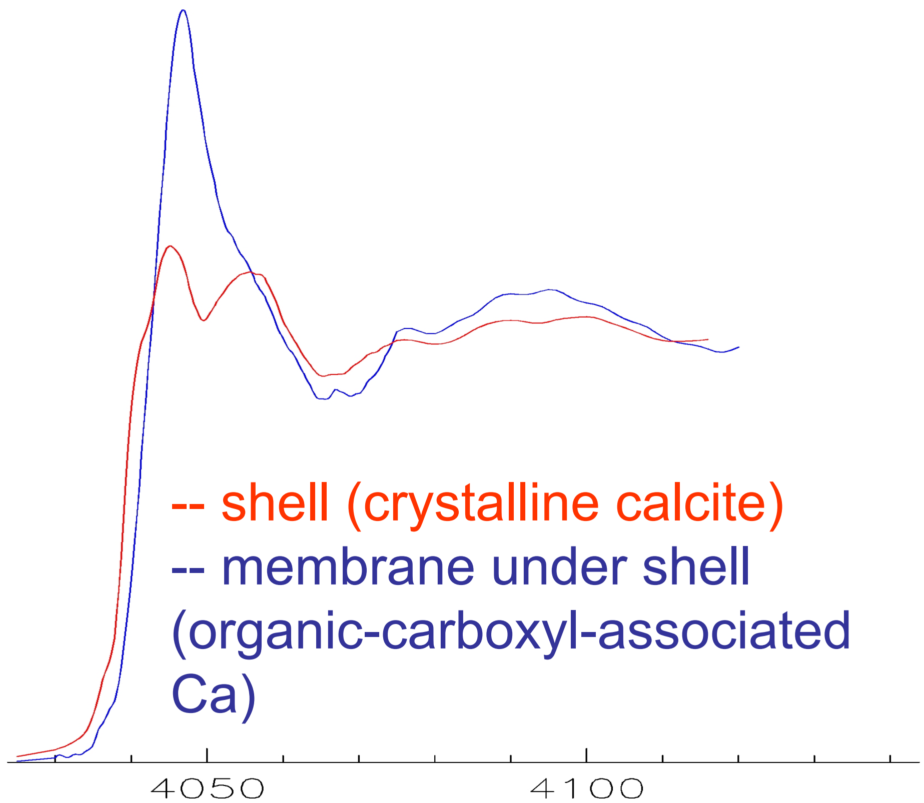




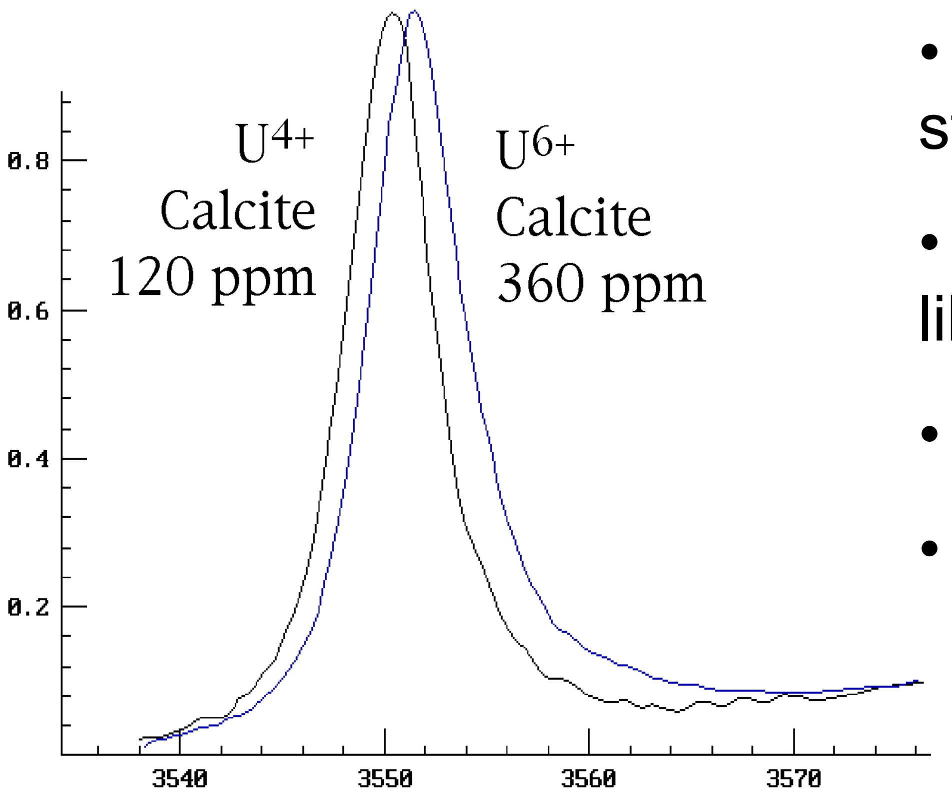
Biomineralization

- Identification of (nano)crystalline and amorphous phases
- The Egg





Uranium M_5 edge:



- Very sensitive to oxidation state
- No interferences from Rb, Sr like at the L_3 edge
- Below Ca K edge
- Sensitivity to ~1ppm

Dual-energy XAS at X15B/TES:

- Dual-energy beam studies, e.g. Fe and S:
    - Selectively allow some harmonic to pass monochromator
    - Requires that beam not be detuned
    - Scan both energies at once, measuring both fluorescence energies independently